

CHARGE NUMBER: 2525
PROJECT TITLE: Chemistry & Isolation of Tobacco
Constituents
PERIOD COVERED: April 4 to May 5, 1980
PROJECT LEADER: S. A. Haut
DATE OF REPORT: May 6, 1980

The polymeric release model, poly- ^{14}C -isopropenyl 2-ethylhexanoate (^{14}C -poly PEX) (in conjunction with Harvey Grubbs, Project 2500) has proven to be a difficult radiochemical synthesis.^{1,4} The polymerization step on the first batch of this material (42mg, 632 μCi) resulted in a considerable loss of the radiolabel in the crude polymer (170 μCi). Some residual activity could be found on the glassware used (<60nCi) but not of sufficient quantity to account for the loss. As a result, a second batch of ^{14}C -PEX monomer was made and purified as before. The mass and activity yields were essentially identical (51mg, 940 μCi). Polymerization of this material also resulted in considerable loss of activity (430 μCi in crude polymer). This time however, condensation was noted on the inner walls of the vessel. When checked for activity this material which was quite volatile contained much activity. At this time we do not know why or how this is occurring.

What polymer we have has been characterized by Mayada L  gue (Project 1703). Altogether, about 50% of the crude combined polymer is of molecular weight 800 and above. At this time, the polymer is being purified by GPC and will be applied to tobacco for smoke studies. GLRC preliminary work is already in progress.

During the past month, five samples were run on the preparative HPLC for Project 2500.

7446-46, 7393-109a
7341-162, 7386-152-1
7341-179

We are currently conducting a trial project exploring the isolation and identifications of components in a browning mixture by TLC and HPLC.⁵ We have achieved the isolation of several materials but their composition or identity as yet

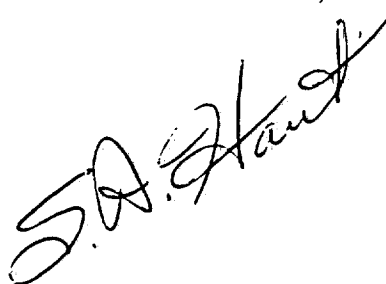
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May 7, 1980

is unknown. GC-MS assistance is being given by Project 0108 on this work and in identifying the by-products from the synthesis of PEX.

References:

1. Barlow, K. R.
2. Core, M. T.
3. Chavis, M. K.
4. Haut, S. A.
5. Edmonds, M. D.

A handwritten signature in black ink, appearing to read "S.A. Haut", is written diagonally across the right side of the references list.

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